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| (21) International Application Number: PCT/GB99/02048 (22) International Filing Date: 8 July 1999 (08.07.99) (30) Priority Data: 9814968.5 10 July 1998 (10.07.98) GB (71)(72) Applicant and Inventor: HINTON, Gerald, Thomas [GB/GB]; Eastfields, Stonebyres, Lanark ML11 9UW (GB). (74) Agent: MURGITROYD & COMPANY; 373 Scotland Street, Glasgow G5 8QA (GB). | | (81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BI, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>Without international search report and to be republished upon receipt of that report.</i> |
| (54) Title: DETERGENT (57) Abstract A detergent composition comprising a granulated percarbonate and a blend which encapsulates the percarbonate is described. The blend comprises a sulphate, carboxymethyl cellulose and a nonionic surfactant. The detergent composition comprises sodium metasilicate and does not include a zeolite, a perborate or a phosphate. The composition is capable of being stored in a water-soluble PVA film packaging for at least nine months and wherein the composition comprises between 1 % and 15 % percarbonate. The composition can include a phosphate substitute such as a polyacrylate. The composition can be compressed into a tablet format and used as a laundering product. | | |

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1 "Detergent"

2

3 This invention relates to a detergent product
4 formulations which can be packaged in water soluble
5 film. The invention also relates to detergent
6 formulations excluding phosphates.

7

8 A product of the invention is ideally for use in the
9 laundering and conditioning of industrial and domestic
10 man-made and/or natural fabrics in semi-automatic or
11 automatic washing machines. It may also be used in
12 dishwashers. For convenience purposes it is useful if
13 this can be achieved by means of a soluble single
14 compartment sachet containing varying amounts of
15 bleaching detergents including for example sodium
16 percarbonate. Typically a sachet may be made from a
17 water soluble film such as PVA.

18

19 Conventional laundering detergents comprise perborates
20 and zeolites and these compounds are not stable in
21 water soluble film. Previous attempts have been made
22 to manufacture fully built detergent and conditioner
23 systems in a sachet have required a twin compartment
24 sachet manufactured from a perforated film. These
25 fully built detergent and conditioner systems generally

1 contain bleaching agents. Sodium percarbonate is
2 recognised in this field as a bleaching agent.
3 However, use of percarbonate in sachets is not popular
4 as it is unstable when combined with other components
5 of a high moisture content.

6
7 Twin compartment sachets have a disadvantage in that
8 they require greater mechanical action to dissolve the
9 sachet and thus have long dispersion times. Also, they
10 are expensive to manufacture.

11
12 Additionally, the perforated film used in these twin
13 compartment sachets does not confer a significant shelf
14 life to the components contained within the sachet,
15 wherein the oxidising power of the bleaching agent is
16 reduced.

17
18 Also, typically these formulations contain zeolites.
19 These have high moisture content which affects the
20 mechanical properties of the film e.g. the pliability.

21
22 Pollution problems arise from the use of phosphates in
23 detergent compositions. Phosphates are required as
24 solubilisers and aid detergency. However, they have a
25 detrimental effect on the environment. Forthcoming
26 European legislation will require the amount of
27 phosphate released into the environment to be
28 minimised.

29
30 It is an object of the present invention to provide a
31 detergent or bleaching agent for cleaning in laundries,
32 or in domestic washing machines or dishwashers which is
33 phosphate free.

34
35 According to one aspect of the present invention there
36 is provided a detergent composition comprising a

1 granulated percarbonate and a blend which encapsulates
2 the percarbonate, the blend comprising a sulphate,
3 carboxymethyl cellulose and a nonionic surfactant,
4 wherein the detergent composition comprises sodium
5 metasilicate and does not include a zeolite, a
6 perborate or a phosphate, and wherein the composition
7 is capable of being stored in a water-soluble PVA film
8 packaging for at least nine months and wherein the
9 composition comprises between 1% and 15% percarbonate.

10

11 Preferably the detergent formulation uses sodium
12 percarbonate, carboxy methyl cellulose, sodium
13 sulphate, a nonionic surfactant blend, sodium silicate
14 and a phosphate substitute, ie an alternative to the
15 phosphates used in detergent formulations.

16

17 The phosphate substitute may be selected from the group
18 comprising silicates, carbonates and polycarboxylates,
19 which should be wholly or substantially anhydrous.

20

21 The invention specifically excludes polyphosphates and
22 preferably includes polyacrylates in powdered or liquid
23 form. In particular formulations the amount of
24 emulsifier metasilicate is also increased and the
25 amount of carboxymethyl cellulose is decreased and
26 bleach activator is added when liquid polyacrylate is
27 used.

28

29 Preferably the detergent further comprises bleach
30 activator such as TAED.

31

32 The detergent may further comprise at least one
33 ingredient chosen from the group comprising linear
34 alkylbenzene sulphonate, sodium lauryl sulphate, sodium
35 carbonate, low foam wetting agent, perfumes, optical
36 brighteners, salts, pigments and enzymes.

1 In one embodiment the detergent formulation is a
2 laundering product.

3
4 In an alternative embodiment the detergent is a machine
5 dishwashing product.

6
7 Suitably the laundry or dishwashing product according
8 to the present invention is packaged in PVA film. A
9 product of the invention is stable in PVA film compared
10 to other products containing zeolites and perborates.

11
12 Suitably the film is 20-80 microns thick.

13
14 The product may be incorporated into a tablet form.

15
16 The granulated form of percarbonate in the above
17 permits efficient bleaching action of the laundry
18 product whilst not effecting the stability of the
19 product in storage.

20
21 While modifications and improvements may be made
22 without departing from the scope of this invention, the
23 following is a description of the invention, with
24 reference to the accompanying diagrams:

25
26 Figures 1a and 1b illustrate a soluble single
27 compartment sachet produced from a polyvinyl alcohol
28 (PVA) film filled with product and heat sealed.

29
30 Figures 2a and 2b illustrate a soluble single
31 compartment sachet produced from PVA film by
32 thermoforming.

33
34 The sachets are sealed such that they contain a laundry
35 and conditioning powder without spillage or air borne
36 contamination which can cause irritation to eyes and/or

1 skin etc.

2

3 Example 1

4

5 The laundry and conditioning powder can be in the form
6 of a super concentrate with a bulk density of not less
7 than 0.75kg/l. The laundry and conditioning powder is
8 preweighed and packed in 50g batches which is
9 sufficient to launder 4.5kg dry weight of mixed fibres
10 (normal soiling) in either hard or soft water
11 conditions.

12

13 In order to determine the storage and durability of
14 sachets containing laundry and conditioner, the sachets
15 were treated as follows:

16

17 1. Laundry and conditioner products including the
18 granulated percarbonate compound were sealed in
19 PVA sachets under atmospheric conditions and
20 stored in various temperatures.

21

22 2. Sachets containing the laundry and conditioner
23 products were sealed in a PVC container under
24 atmospheric conditions as stored at various
25 temperatures.

26

27 The samples of both 1 and 2 above were stored for nine
28 months whereupon they were added to separate washing
29 cycles. In both cases the samples were found to be
30 stable (both before use and after storage) with no
31 deterioration of the product or the sachet containing
32 the product.

33

34 Sachets were dissolved in cold water (20°C) using a
35 combination of water flow and mechanical agitation
36 whereupon sachets and contents were typically

1 completely dissolved with no residue within 90 seconds.

2

3 The polyvinyl alcohol film was 30-85 microns (+/- 10-
4 15%) thick. The polyvinyl alcohol film is both
5 biodegradable and nonhazardous.

6

7 The process for producing the sachets according to
8 figures 1a and b containing the dishwashing, laundry
9 and/or conditioner product requires a form filling
10 machine modified such that the sachet is produced with
11 a minimum number of folds and seals.

12

13 Alternatively thermoforming of film can be used to
14 produce filed sachets as illustrated in Figure 2.

15

1 Example 2

2

3 Typical detergent product formulations

4

| | | |
|----|---------------------------------------|-------------|
| 5 | Linear alkylbenzene sulphonate (LABS) | 0.5% |
| 6 | Sodium Percarbonate | 1-15% |
| 7 | Carboxy Methyl Cellulose (CMC) | 1-5% |
| 8 | Sodium Sulphate Anhydrous | 5-35% |
| 9 | Sodium Carbonate | 0-35% |
| 10 | Nonionic Surfactant Blend | 1-10% |
| 11 | Low Foam Wetting Agent | 0-2% |
| 12 | Sodium Metasilicate | 1-30% |
| 13 | Perfumes | 0-1.5% |
| 14 | Optical Brighteners | 0-1% |
| 15 | Salts | 0-10% |
| 16 | Enzymes (blended) | 0-5% |
| 17 | Copolymer | 0-10% |
| 18 | Water Soluble Dye Pigment | 0-2% |
| 19 | Sodium Polyacrylate | 1-30% |
| 20 | (eg Sandoperol) | (preferably |
| 21 | | 2-12%) |
| 22 | Bleach Activator | 0.1-2% |

23

24 Minor ingredients as required.

25

26 Linear alkylbenzene sulphonate may be replaced with 0.5
27 to 10% sodium lauryl sulphate to produce a more
28 environmentally friendly product.

29

30 Varying amounts of the above components may be used
31 depending on the type of product required, i.e. for
32 laundering, dishwashing or conditioning.

33

34 In the following examples nonoinic surfactant blend and
35 low foam wetting agent are together referred to as
36 liquid blend.

1 Example 3

2

3 Laundry Product using Liquid Polyacrylate

4

5 A laundering product was prepared and packaged in PVA
6 film.

7

8 The formulation consisted of

9

| | | |
|----|------------------------------|------|
| 10 | Linear alkylbenzene sulphate | 2% |
| 11 | Sodium Percarbonate | 3% |
| 12 | Carboxy Methyl Cellulose | 1% |
| 13 | Sodium Sulphate Anhydrous | 25% |
| 14 | Sodium Carbonate | 28% |
| 15 | Liquid blend | 2% |
| 16 | Sodium Metasilicate | 30% |
| 17 | Anionic Sodium Polyacrylate | 10% |
| 18 | Perfumes | 0.8% |
| 19 | Optical Brighteners | 0.5% |
| 20 | Salts | 2% |
| 21 | Enzymes (blended) | 1.5% |
| 22 | Copolymer | 2% |
| 23 | Bleach Activator (TAED) | 1% |

24

25 Prior to mixing the liquid polyacrylate is blended with
26 approximately 60% of the anhydrous sulphate and dried.

27

28 Alternatively, the polycrylate may be mixed with any of
29 the powdered materials, dispersed through the powder.

30

31 Example 4

32

33 Laundry Product

34

35 A laundering product was prepared and packaged in PVA
36 film.

1 The formulation consisted of

2

| | | |
|----|------------------------------|-------|
| 3 | Linear alkylbenzene sulphate | 2% |
| 4 | Sodium Percarbonate | 5.1% |
| 5 | Carboxy Methyl Cellulose | 1.0% |
| 6 | Sodium Sulphate Anhydrous | 20% |
| 7 | Sodium Carbonate | 33.0% |
| 8 | Liquid blend | 2.0% |
| 9 | Sodium Metasilicate | 30% |
| 10 | Perfumes | 0.8% |
| 11 | Optical Brighteners | 0.5% |
| 12 | Salts (NTA Powder) | 2% |
| 13 | Enzymes (blended) | 1.5% |
| 14 | Copolymer | 2.0% |
| 15 | Bleach Activator (TAED) | 1% |

16

17 Inclusion of copolymer improved redeposition.

18

19 Production of Formulation

20

21 The percarbonate was added to the sachet as shown in
22 Figure 1 in the form of granules. These granules
23 comprised percarbonate, sulphate and carboxy methyl
24 cellulose in varying amounts together with a blend of
25 nonionic surfactants to create a binding agent. These
26 components were processed in order to produce a dust
27 free granule of a diameter not less than 150 microns.

28

29 In order to produce the granules a horizontal type
30 mixer was used. A liquid blend of the abovementioned
31 laundry components was added to the mixer from a high
32 pressure vessel incorporating an agitator. The liquid
33 blend was fed in at a pressure of 60 pounds per square
34 inch.

35

36

1 The finished granulated detergent is fully
2 biodegradable and has a stable pH range of 10-11, which
3 does not affect the PVA film stability as used in this
4 invention.

5
6 Trials have shown that using nonionic surfactants
7 comprising alkyl aryl polyglycol ethoxylates through
8 the alkyl group C₆₋₁₂ (typically C₈₋₁₀) is stable and
9 gives the best results even after storage in excess of
10 9 months.

11
12 A typical encapsulation blend is as follows:

13
14 Sodium Sulphate (Anhydrous) 5-98%
15 Carboxy Methyl Cellulose 1-25%
16 Nonionic Surfactant blends 1-40%

17
18 Alternative nonionic surfactant blends comprising
19 alcohol polyglycol ethoxylate oxide in the range of
20 0.5-5% have been used successfully.

21
22 The advantages of the invention and of the ways in
23 which the disadvantages of the previously known
24 arrangements are overcome include encapsulation of a
25 percarbonate with a powder/liquid blend forming a
26 granular product of suitable size and strength for use
27 in a hot or cold process.

28
29 A single component sachet sealed such that the
30 percarbonate does not decompose in the detergent
31 contained within the sachet.

32
33 Upon dissolution the PVA leaves no residues i.e. it is
34 fully dissolved.

35
36

1 No mechanical action is required to dissolve the PVA
2 film.

3
4 The encapsulation process extends the shelf life of
5 fully built detergent within the PVA sachet.

6
7 In the super concentrated form, a laundering
8 formulation normally requires 50g per 4.5kg (dry
9 weight) wash with normal soiling.

10

11 Example 5(a)

12

| | | | |
|----|-----------------------------------|-----|---|
| 13 | Sodium Sulphate | 20 | % |
| 14 | Sodium Carbonate | 40 | % |
| 15 | Sodium Metasilicate Anhydrous | 15 | % |
| 16 | Sodium Percarbonate Peroxyhydrate | 10 | % |
| 17 | CMC | 1 | % |
| 18 | Alcohol Polyglycol Ethoxylate | | |
| 19 | Oxide + Cationic Resin Dye | | |
| 20 | Transfer Inhibitor | 5 | % |
| 21 | LABS | 1.2 | % |
| 22 | NTA Powder/EDTA Powder | 5 | % |
| 23 | Enzymes (optional) | 0.5 | % |
| 24 | Co Polymer | 1 | % |
| 25 | TAED | 1 | % |
| 26 | OBA (optional) | 0.3 | % |
| 27 | Perfume (optional) | - | |

28

29 Example 5(b)

30

| | | | |
|----|-------------------------------|----|---|
| 31 | Sodium Sulphate | 14 | % |
| 32 | Sodium Carbonate Dense | 50 | % |
| 33 | Sodium Metasilicate Anhydrous | 10 | % |
| 34 | Alcohol Polyglycol ether + | | |
| 35 | Cationic resin Dye | | |
| 36 | Transfer Inhibitor | 10 | % |

| | | |
|---|--------------------|------|
| 1 | CMC | 2 % |
| 2 | Percarbonate | 4.5% |
| 3 | Optical Brightners | 0.3% |
| 4 | LABS | 1.2% |
| 5 | NTA Powder (salts) | 5 % |
| 6 | Perfumes | 0.5% |
| 7 | Enzymes | 0.5% |
| 8 | Co Polymer | 1 % |
| 9 | TAED | 1 % |

10

11 In the above Examples, no phosphate or phosphate
12 substitute is used. The level of sodium carbonate is
13 increased in Example 5(b) to act as a filler and
14 provides the alkalinity previously provided by
15 phosphate. An increased level of percarbonate acts
16 similarly.

17

18 Example 6

19

| | | |
|----|-----------------------------------|------|
| 20 | Sodium Metasilicate | 18 % |
| 21 | Sodium Carbonate | 40 % |
| 22 | Sodium Sulphate | 14 % |
| 23 | Sodium Polyacrylate Acid | 6 % |
| 24 | Sodium Percarbonate | 8 % |
| 25 | CMC | 1 % |
| 26 | NTA/EDTA Powder | 4 % |
| 27 | LABS | 2 % |
| 28 | Perfume | 0.8% |
| 29 | Liquid Alcohol Polyglycol Ether + | |
| 30 | Dye Transfer Inhibitor | 3 % |
| 31 | Enzymes | 0.6% |
| 32 | OBAs | 0.3% |
| 33 | Co Polymer | 1 % |
| 34 | TAED | 1.3% |

35

36

1 **CLAIMS**

2

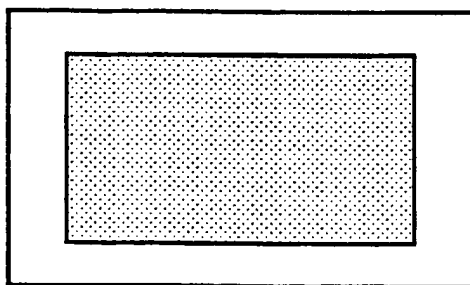
- 3 1. A detergent composition comprising a granulated
4 percarbonate and a blend which encapsulates the
5 percarbonate, the blend comprising a sulphate,
6 carboxymethyl cellulose and a nonionic surfactant,
7 wherein the detergent composition comprises sodium
8 metasilicate and does not include a zeolite, a
9 perborate or a phosphate, and wherein the
10 composition is capable of being stored in a water-
11 soluble PVA film packaging for at least nine
12 months and wherein the composition comprises
13 between 1% and 15% percarbonate.
- 14
- 15 2. A composition as claimed in claim 1 wherein the
16 percarbonate is sodium percarbonate.
- 17
- 18 3. A composition as claimed in claim 1 or claim 2
19 wherein the sulphate is sodium sulphate.
- 20
- 21 4. A composition as claimed in any of the preceding
22 claims wherein the surfactant is alkyl (C₆ to C₁₂)
23 aryl polyglycol ethoxylate.
- 24
- 25 5. A composition as claimed in any of the preceding
26 claims wherein the composition further comprises
27 at least one of the ingredients chosen from the
28 group comprising linear alkylbenzene sulphonate,
29 sodium carbonate, low foam wetting agent,
30 perfumes, cationic surfactant, optical
31 brighteners, salts and enzymes.
- 32
- 33 6. A composition as claimed in any one of the
34 preceding Claims wherein the composition further
35 includes a phosphate substitute.
- 36

- 1 7. A composition as claimed in Claim 6 wherein the
2 phosphate substitute is selected from the group
3 comprising silicates, carbonates and
4 polycarboxylates.
5
- 6 8. A composition as claimed in Claim 7 wherein the
7 phosphate substitute is a polyacrylate.
8
- 9 9. A composition as claimed in Claim 8 wherein the
10 phosphate substitute is sodium polyacrylate.
11
- 12 10. A composition as claimed in any of the preceding
13 claims wherein the composition is a laundering
14 product.
15
- 16 11. A composition as claimed in any of the preceding
17 claims wherein the composition is a machine
18 dishwashing product.
19
- 20 12. A composition as claimed in any one of the
21 preceding claims wherein the PVA film is 20-80
22 microns thick.
23
- 24 13. A composition as claimed in any one of the
25 preceding claims wherein the product is compressed
26 into a tablet format.
27

$2/2$

Figure 2

(a)



(b)

